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First Bimonthly Progress Report

NEW CONCEPT STUDIES, CW DEFENSE

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ABSTRACT

An experimental apparatus for vapor phase adsorption experiments has been assembled. Preliminary runs on the apparatus gave uniform saturation of the gas air stream with organic compounds and water. The vapor phase chromatographic apparatus to be used in the experimental work was checked on highly dilute samples of ethylthiophosphate. Based on a literature survey on experimental methods of determining wetting and spreading characteristics of sorbents, an apparatus for contact angle and adhesion tension measurement was designed and is being built to order.

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I. OBJECTIVE

The objective of this work is to study the basic physicochemical phenomena that determine the sorption rates and equilibria.

II. EXPERIMENTAL RESULTS

A. VAPOR PHASE ADSORPTION

The laboratory work this period was entirely directed toward the design, assembly, and start-up of a vapor phase adsorption unit. The adsorption apparatus consists of three sections: the feed mixing section, the adsorption section, and the analysis and removal section. The apparatus was completely assembled and instrumented to permit us to carry out experiments on adsorption from the vapor phase at temperatures from -25 to +125°F with an air stream containing up to a maximum concentration of CW agent and water corresponding to their vapor pressures at the temperature of adsorption. Several preliminary runs on the unit with non-toxic liquids showed the necessity of a few modifications to permit a completely safe and smooth operation of the unit (by-passing of the adsorber tube, sampling of the gas stream before and after adsorption by alternate chemical and instrumental methods, installation of canisters for final adsorption). The apparatus is described in detail in Appendix I. Preliminary experiments to determine the saturating efficiency and to provide uniform saturation are discussed in detail in Appendix II.

Preliminary vapor phase chromatographic work on highly diluted ethylthiophosphate showed that this compound can be detected with a hydrogen flame detector in quantities of 0.005µg, which corresponds to a concentration of about 0.25µg/l. A much higher sensitivity will be attained with an electron capture detector. Information from Perkin-Elmer Co. indicates that the sensitivity of electron capture detectors is about 100-fold greater for chlorine-containing compounds, and about 10- to 30-fold greater for fluorine compounds than the hydrogen flame detector is for hydrocarbons. An Aerograph Model 600B chromatograph with both detectors to be used exclusively on this project is on order and delivery is expected shortly. The analytical reagents to be used for the chemical analysis of these agents were secured.

B. LIQUID PHASE ADSORPTION

The preliminary literature survey on experimental methods of determining wetting and spreading characteristics of solvents with organic materials was extended. An apparatus for determining adhesion tension and contact angles has been designed and is on order.

III. PROGRAM

A. VAPOR PHASE ADSORPTION

The first experiments will be carried out on Whetlerite feed stock obtained from Pittsburgh Chemical Company. Other adsorbents are being secured.

The variables to be studied in our experimental program are:

- (a) Concentration of CW agent
- (b) Humidity
- (c) Flow rate
- (d) Particle size of adsorbent
- (e) Temperature

A preliminary investigation using factorial design will determine which variables are significant for testing. In the initial experiments, only the effects of variables (b), (c), and (d) will be investigated since the results must be strongly affected by any change in variables (a) and (e), the concentration of the CW agent and the temperature of the adsorption process. A factorial design that involves these three factors at two levels for each will be used. A series of eight experimental runs will give a better estimate of the true difference in means and will permit the estimation of interaction effects. A further extension of the experimental design by "confounding" will reduce the number of experiments and permit us to include further variables and disregard nonsignificant variables. These experiments will yield results expressed in the form of adsorptive capacity at the break point and total adsorptive capacity.

The extension of the work, including concentration variation will determine the adsorption isotherms and permit their calculation, and the extension including temperature variation will permit the calculation of the heat of adsorption. In this stage of the work, regression analysis will be used to develop quantitative relationships among the important variables. In these additional runs, the important variables will be examined at several levels so that more exact relationships can be determined. A detailed experimental program for this phase of the work has been prepared.

B. LIQUID PHASE ADSORPTION

Model experiments on capillary action and rate of penetration of liquid agents, and on determination of retentive capacity will be started as soon as the vapor phase adsorption unit is in full operation.

APPENDIX I. DESCRIPTION OF APPARATUS

The dynamic adsorption apparatus consists essentially of a flow system in which an air stream is separately saturated with water and CW agent, then passed through a bed of the adsorbent whose adsorptive capacity for CW agent is being studied. A schematic diagram of the flow system is shown in Figure 1.

As shown in this diagram, cylinder air regulated to about two psig is filtered through glass wool and dried with caustic pellets and Drierite in F-1. The dry, clean air is metered through rotameter R-1 and then is subdivided three ways. One stream passes through another rotameter, R-2, and on to a fritted-glass sparger, S-1, in a constant temperature bath. This sparger or bubbler will contain water to saturate the air stream to various levels. Another stream is metered by R-3 and then saturated with CW agent in S-2, which is also in a constant temperature bath. The third stream, whose flow rate is known by difference, passes directly to a mixing cross where all three stream converge and are blended together.

The total stream then passes to the adsorber, A-1, containing adsorbent material under study. Since adsorption studies will be made in the temperature range -25 to +125°F, the adsorber is surrounded by a constant temperature double-walled bath capable of operating in this total range. Ethylene glycol will be used in the bath, and the annular space between the two walls will be filled with a freezing mixture or insulation.

After adsorption, the flow stream will be analyzed for residual CW agent either with a special vapor phase chromatograph that is fitted with both flame ionization and electron capture detectors or with a colorimetric analysis requiring the use of bubblers containing an absorbing solution. A by-pass line around the adsorber has been installed to allow analysis of the total stream before adsorption.

All tail gases from the flow system will be passed through a gas mask canister before being passed to the hood stack. In this way, only pure air should be vented by the hood. As a further precaution an adsorbing filter will be installed in the hood blower suction line.

Some additional details shown in the Figure are:

1. STV (Solenoid Trip Valve). This is actuated by a high pressure at manometer PI-1 and will shut down the system in case of plugged flow.
2. CV (Check Valve). This will prevent accidental flow of water-saturated air into the CW saturator with consequent hydrolysis of the CW agent.
3. S-2 (CW Saturator). This device was provided by the Army. It uses a cylindrical alundum wick to saturate the air stream with CW agent. A cylindrical glass baffle causes the air stream to contact the wick on both inner and outer surfaces.

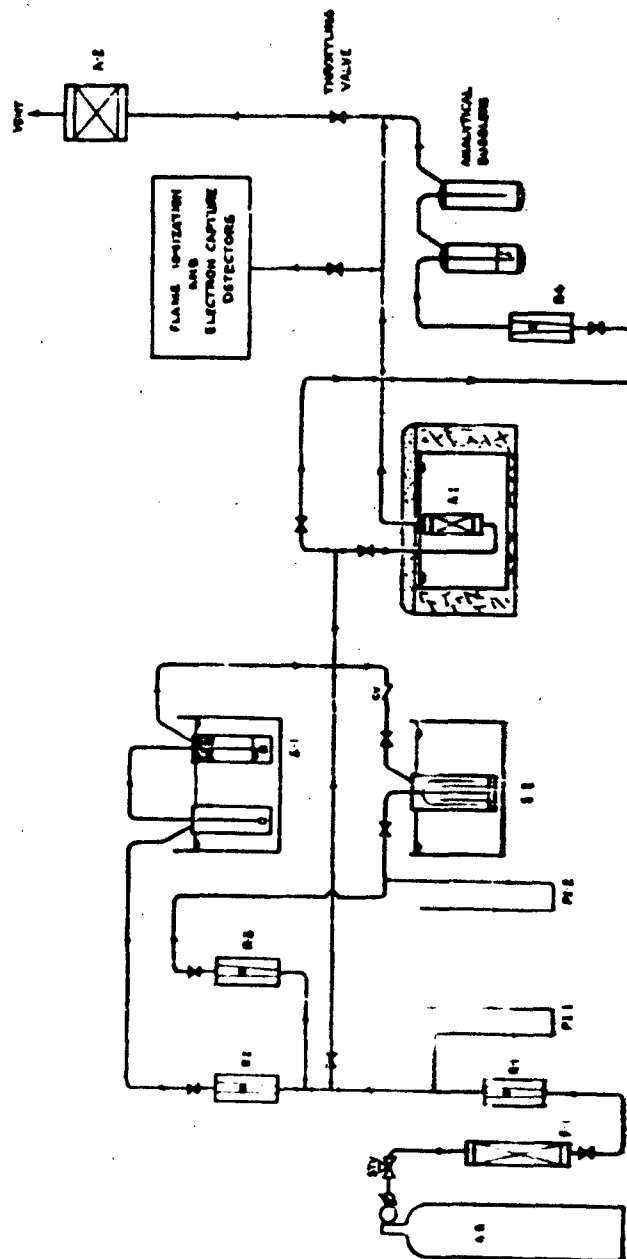


Figure 1. Schematic Drawing of Dynamic Absorption System.

The objective of the saturation equipment is to insure that the total air stream after mixing and before adsorption is from 50-80% humid with respect to water and 100% (or nearly so) humid with CW agent, both at the adsorption temperature which may vary from -25 to +125°F

APPENDIX II. SUMMARY OF PRELIMINARY SATURATION
EXPERIMENTS

Experiments were run to become acquainted with the saturating efficiency of the device supplied by the U. S. Army for contacting flow streams with CW agent and also to try out a fritted glass bubbler of our own design. Table 1 summarizes this work and is largely self-explanatory.

The outcome of these tests was that the Army device was considered to be the more satisfactory design for our purposes. The need for good insulation and, in some cases, heating on the small lines leaving the saturator was indicated.

Table 1
TESTING EFFICIENCY OF SATURATING DEVICES

Run No.	PURPOSE	Time Elc.	Air Flow Rate 24/hr	Satur- ation Temp °F	Satura- tion Pressure mm Hg	Absolute Humidity lb/lb dry air	Total Toluene Pick-up 1000 Set lb	Trioluene on Adsorber lb	Relative Humidity %	Observations and Conclusions
1	Test our own saturator	577	25.8	24	-	0.14	2.08	1.69	81.2	Try Army saturator
2	Test Army saturator	191	25.1	42	-	0.31	1.487	0.3231	22.1	Condensation in line after saturator
3	Try lower saturation temperature	166	60.1	25	-	0.13	1.289	0.7959	61.0	Still condensing in line
4	Try with same insulation	124	60.8	25	-	0.13	0.980	0.524	53.5	Need heat on lines
5	Try with heated, insu- lated off-take lines	120	60.4	25	-	0.13	0.942	0.848	90.0	Still condensing slightly
6	Try with more heat and insulation	120	60.4	26.5	-	0.135	0.978	0.904	92.5	Need insulation on saturator head
7	Try at higher temperature	123	56.3	30.5	-	0.165	1.142	0.944	82.3	Efficiency worse at higher temp.
8	Effluent saturated a.r. with 20-ppm air to avoid con- densation. Also measure saturation pressure	105	58.2	31.5	4.1	0.176	1.076	0.9317	92.3	Satisfactory
9	Repeat experiment above at slightly higher tem- perature	120	59.3	36	4.0	0.201	1.572	1.1170	71.0	Possible b.d. data. Repeat.
10	Try lower flow rate	120	22.0	26.3	5.0	0.224	0.592	0.5400	91.2	Satisfactory run

<p>AD Scientific Research Corp., Everett, Wash. NEW CONCEPT STUDIES, Dr. EUGENE-J. O. Smith, B. R. Pines, and D. A. Duncan Blanchard, Dept. No. 1, 1 July-1 Sep 63, P. Illus. 149, 150 Contract No. DA18-108-AMC-238 (A) Order No. GPO-21960</p> <p>An experimental apparatus for vapor phase adsorption experiments has been assembled. Preliminary runs on the apparatus gave uniform saturation of the gas air stream with organic compounds and this is to be used in the experimental work. The apparatus is based on a literature survey on experimental methods of determining wetting and spreading characteristics of sorbents, an apparatus for contact angle and adhesion tension measurement was designed and is being built to order.</p>	<p>UNCLASSIFIED</p> <p>1. ON Agent Sorption 2. Contract DA18-108-AMC-238 (A)</p>	<p>UNCLASSIFIED</p> <p>1. ON Agent Sorption 2. Contract DA18-108-AMC-238 (A)</p>	<p>UNCLASSIFIED</p> <p>1. ON Agent Sorption 2. Contract DA18-108-AMC-238 (A)</p>
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